Effects of Mn addition on microstructures and mechanical properties of 10Cr ODS ferritic/martensitic steels

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1. Introduction

With a recent accelerated evolution of nuclear reactors combined with ever-increasing world energy demands, Generation IV future nuclear systems have spurred considerable research and developmental interests in several countries with nuclear power reactors because it is believed that the Gen IV improves efficiency, safety, reliability, and proliferation-resistance of nuclear reactors [1]. To achieve them, improved core structural materials with a potential to be applicable at elevated temperature under severe neutron exposure environment are required. Ferritic/martensitic (FM) steels are very attractive for the structural materials of fast fission reactors such as a sodium cooled fast reactor (SFR) owing to their excellent irradiation resistance to a void swelling [2,3], but are known to reveal an abrupt loss of their creep and tensile strengths at temperatures above 600 °C [4]. Accordingly, high temperature strength should be considerably improved for an application of the FM steel to the structural materials of SFR. Oxide dispersion strengthened (ODS) FM steels are considered to be promising candidate materials for high- temperature components operating in severe environments such as nuclear fusion and fission systems due to their excellent high temperature strength and radiation resistance stemming from the addition of extremely thermally stable oxide particles dispersed in the ferritic/martensitic matrix [5-7]. To develop an advanced ODS steel for core structural materials for next generation nuclear reactor system applications, it is important to optimize its compositions to improve the high temperature strength and radiation resistance.

This study investigates effects of Mn addition on microstructures and mechanical properties of 10Cr ODS FM steel. For this, two 10 Cr ODS FM steels were prepared by mechanical alloying (MA), hot isostatic pressing (HIP), and hot rolling process. Tensile tests were carried out at room temperature and 700 °C to evaluate the influences of the Mn element on the mechanical properties. The microstructures were observed using SEM, electron back-scatter diffraction (EBSD) and transmission electron microscopy (TEM) with energy dispersive spectroscopy (EDS).

2. Experimental procedure

The work presented here was focused on ODS FM steels, the chemical compositions of which are given in Table 1. The ODS FM steels, sample A: Fe-10Cr-1Mo and sample B: Fe-10Cr-1Mo-0.6Mn in wt% were fabricated by MA and HIP processes.

Table 1. Chemical composition (wt. %) of 10Cr-1Mo ODS FM steel.

Alloy (wt.%)	Fe	Cr	Mo	Y_2O_3	Mn
A : 10Cr-1Mo	Bal.	10	1.2	0.35	-
B : 10Cr-1Mo-0.6Mn	Bal.	10	1.2	0.35	0.6

Pre-mixed metallic raw powders and yttria powder were mechanically alloyed by a horizontal ball-mill apparatus, CM-08, under a high purity Ar gas (purity in 99.999%) atmosphere. The mechanical alloying was performed at an impeller rotation speed of 300rpm for 40hrs with a ball-to-powder weight ratio (BPWR) of 10:1. MA powders were then placed in an AISI 304 L stainless steel containers. The sealed capsules were degassed at 500 ^oC below 5×10^{-3} torr for 1h. The HIP was carried out at 1150 ^oC under a pressure of 100 MPa for 4 hr at a heating rate of 5^oC/min and followed by furnace cooling. Hipped samples were hot rolled in a fixed rolling direction for a plate shape, which resulted in a final reduction rate of 65%, and then normalized at 1150 ^oC for 1hr and tempered at 750 ^oC for 1hr through air cooling respectively. To evaluate mechanical property, according to ASTM E8, tensile tests were conducted at room temperature and 700 ^oC in air at a strain rate of $3.3x10^{-4}s^{-1}$. Microstructure of the tempered plate was characterized by an optical microscopy. Samples for the optical microscopy were mechanically wet ground and chemically etched in 5% aqua regia solution for 10min. The grain morphology was observed by SEM and EBSD after the electro-polishing in a 5% $HClO_4$ + 95% methanol solution in vol. % at 18V with 0.5mA at -50 °C. To examine the size distribution and the elemental analyses on the precipitates, TEM observation with EDS was carried out. For this, the carbon extraction replicas were prepared by means of a mechanical polishing, etching with a mixed solution of 93 vol.% water, 5 vol.% nitric acid and 2 vol.% fluoric acid, a carbon coating, and removing the replicas by electrochemical etching with a mixed solution of 90 vol.% methanol and 10 vol.% hydrochloric acid.

3. Results and Discussions

The tensile test results of the 10Cr ODS FM steels at room temperature and 700 ^{o}C are shown in Fig. 1. It was found that sample B shows a higher strength than that of sample A at room temperature. At an elevated temperature of 700 ^{o}C, on the other hand, sample B has a comparable strength with sample A. At room temperature, the yield and tensile strengths and the elongation of sample A were measured to be 880 MPa, 1043 MPa and 11%, respectively. In the case of sample B, the yield and tensile strengths and the elongation were determined to be 917 MPa, 1081 MPa and 10.1%, respectively. At 700 ^{o}C, the yield and tensile strengths and the elongation of sample A were measured to be 200 MPa, 236 MPa and 27%, respectively. In the case of sample B, the yield and tensile strengths and the elongation were determined to be 192 MPa, 231 MPa and 29%, respectively. These results indicate that tensile properties of 10Cr ODS FM steel can be improved by Mn addition.

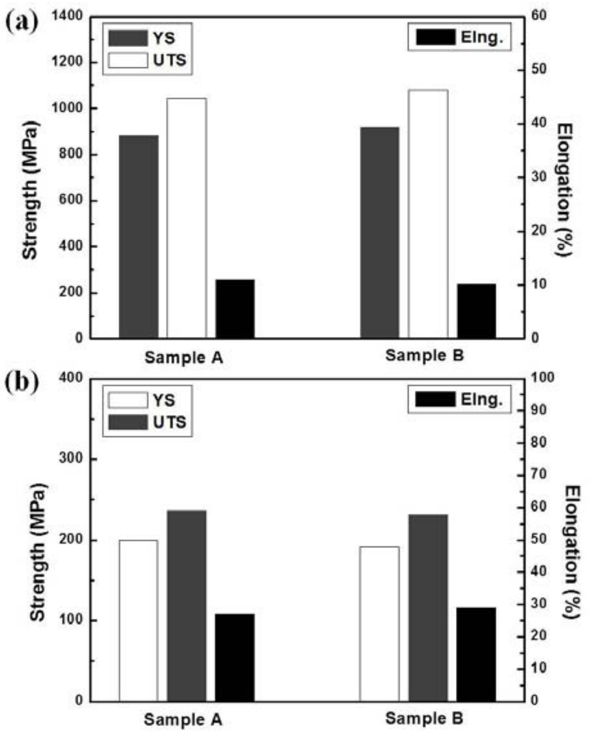

Fig. 1. Tensile test results of 10Cr-1Mo and 10Cr-1Mo-0.6Mn ODS FM steels in the temperature (a) RT and (b) 700 ^{o}C.

Optical microstructures of sample A (10Cr-1Mo) and sample B (10Cr-1Mo-0.6Mn) ODS FM steels are shown in Fig. 2. The results clearly show that both sample A and B consist of fine equiaxed grains and a small portion of elongated grains along with the hot rolling direction. Considering the fact that the temperature is increased from room temperature to 1150^{o}C for the HIP process, it is thought that fine equiaxed grains are martensite and the elongated grains parallel to the rolling direction are delta-ferrite, which reside untransformed without transforming into gamma during the rise of temperature. This is responsible for the presence of a regionally high concentration of ferrite former, Cr and Mo [8]. When a portion of fine equiaxed grains between sample A and B are compared, it is found that sample B has more equiaxed grains than sample A, suggesting that Mn affects the microstructure change.

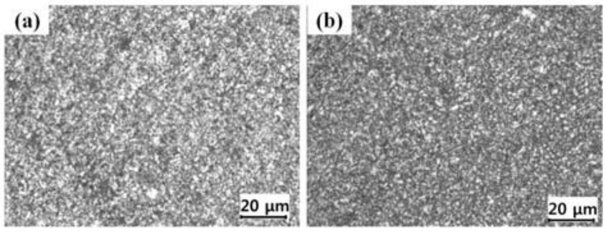

Fig. 2. Optical micrographs of (a) 10Cr-1Mo and (b) 10Cr-1Mo-0.6Mn ODS FM steels.

Fig. 3 shows the SEM micrographs of the grain morphology for 10Cr ODS FM steels: (a,b) sample A and (c,d) sample B. It can be seen that both sample A and B have martensite structures with finely dispersed carbides along the grain boundaries, as well as delta-ferrite structures. It should be note that the areal fraction of martensite structures in sample B with 0.6 wt% Mn is increased and the equiaxed grains in size become finer, implying that the formation of martensite is enhanced by addition of Mn acting as austenite former.

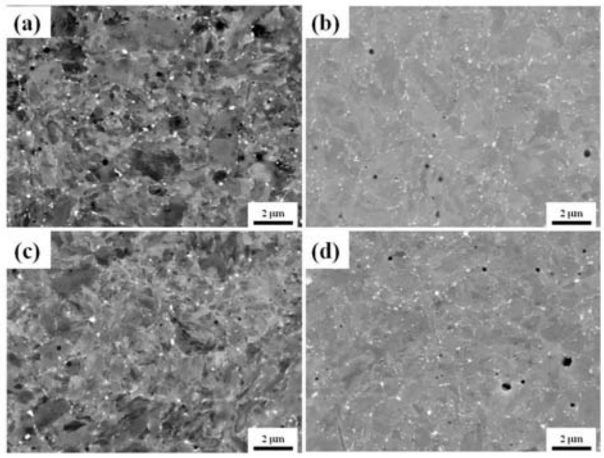

Fig. 3. SEM micrographs (a) 10Cr-1Mo and (b) 10Cr-1Mo-0.6Mn ODS FM steels.

The TEM images with EDS of the precipitates and oxide particles taken from the replica samples of 10Cr ODS FM steels are exhibited in Fig. 4. In sample A, the precipitate was found to be mainly $M_{23}C_6$ (M=Fe, Cr and Mo) formed in the grain boundaries. In the case of sample B, it was found that Mn was dissolved in $M_{23}C_6$ (M=Fe, Cr and Mo). Based on the observation that a number of precipitates in sample B are slightly larger than in sample A, even if there is a small portion of precipitate coarsening, it is considered that the carbide formation is instigated by the Mn addition, resulting in the grain refining effect. Coupled with the increase of martensite structures, this result enable the conclusion that the improvement of tensile properties seen in sample B is attributed to the microstructure change by the addition of Mn. In addition, it can be seen that oxide particles are uniformly distributed in all ODS FM steels. The complex oxides were identified to be Y-Cr-Ti-Zr-O and Y-Cr-Ti-O in sample A and Y-Cr-Ti-Zr-O and Cr-Ti-Zr-O in sample B, respectively.

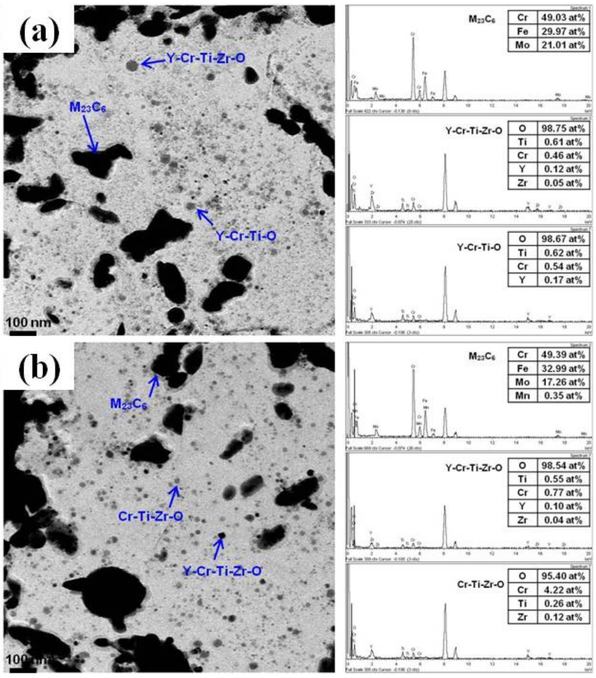

Fig. 4.TEM/EDS results of precipitates and oxide particles in (a) 10Cr-1Mo and (b) 10Cr-1Mo-0.6Mn ODS FM steels.

4. Conclusions

In the present study, the effects of Mn addition on the microstructure and mechanical properties of ODS FM steels were investigated. The ODS FM steels were manufactured by the MA, HIP and hot-rolling processes. The addition of Mn led to the increase of martensite structures with a relatively higher density of precipitates, resulting in the improvement of mechanical properties. These preliminary results will be helpful in the development of advanced ODS steel.

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